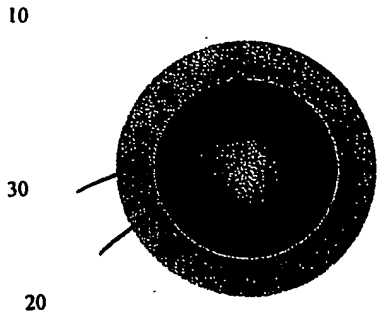


FIG. 1A

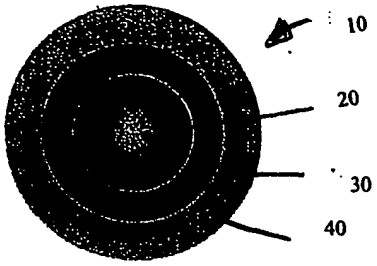
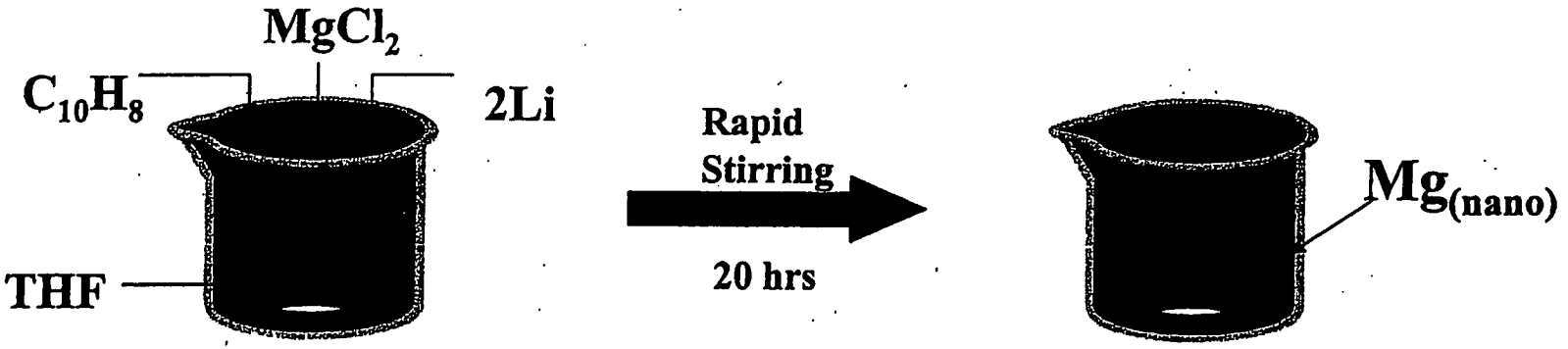


FIG. 1B

Mg Synthesis

1. Synthesis (performed in an Ar Glove Box)



2. Remove THF with dissolved by-products

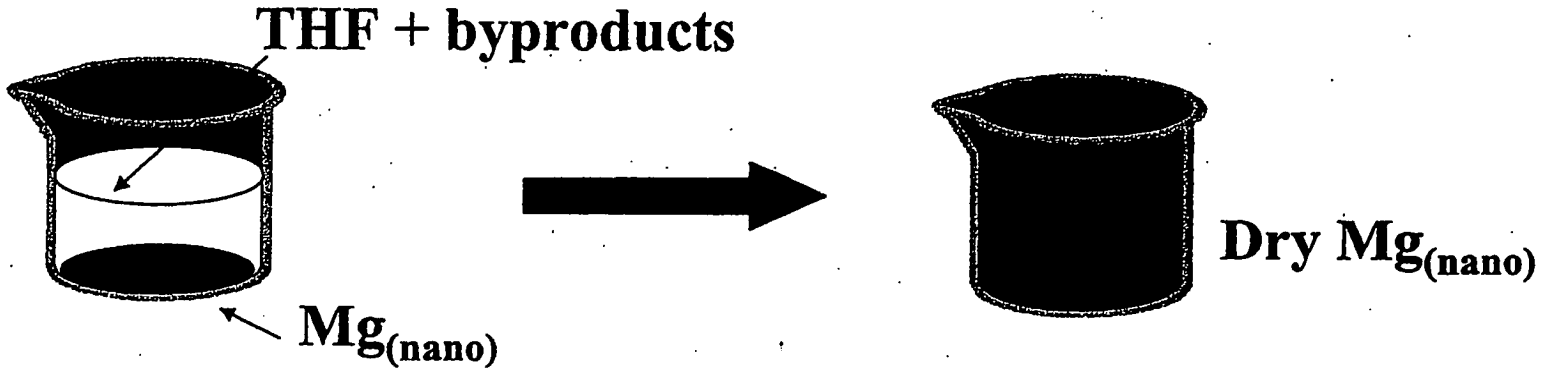


FIG. 2

Synthesized Mg

FIG. 3

Sample	Amount Made	XRD crystallite size (Scherrer equation)*
Mg 1-2	0.198g	23nm
Mg 1-10	0.4g	Used in Pd capping
Mg 1-13	0.4g	37nm
Mg 1-18	0.2g	34nm
Mg 1-41	0.2g	Used for Co capping
Mg 1-51	0.4g	33nm
Mg 1-57a	0.5g	25nm
Mg 1-57b	0.5g	Still in dry box
Mg 1-57c	0.5g	Almost amorphous

$$*t = [(0.9)(0.154)]/[(\beta)(\cos\theta)]$$

t = crystallite size in nm

β = full width half max

θ = Bragg angle

Klug and Alexander, 1950

Title: HYDROGEN STORAGE MATERIAL BASED ON A MULTILAYERED CORE/SHELL STRUCTURE
Inventor's Name: Peter C. EKLUND, et al.
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As Synthesized Mg XRD

XRD of Mg 1-13 sample matches that of an indexed Mg pattern
Material is coated with amorphous sp^2 carbon allowing it to be stable in air

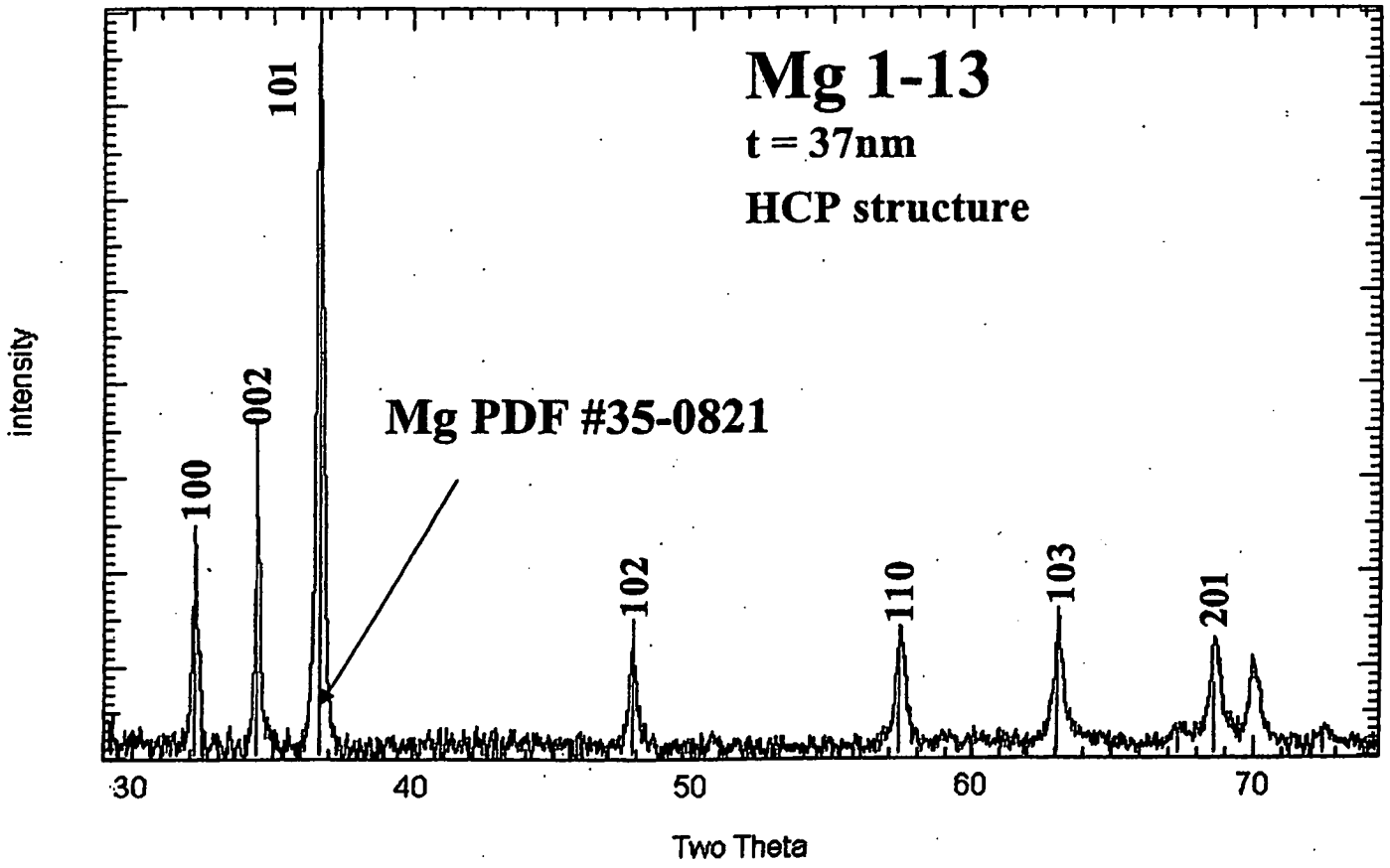


FIG. 4

Still Mg Metal!

Mg 1-13 sample was stable in atmospheric conditions for 3 months

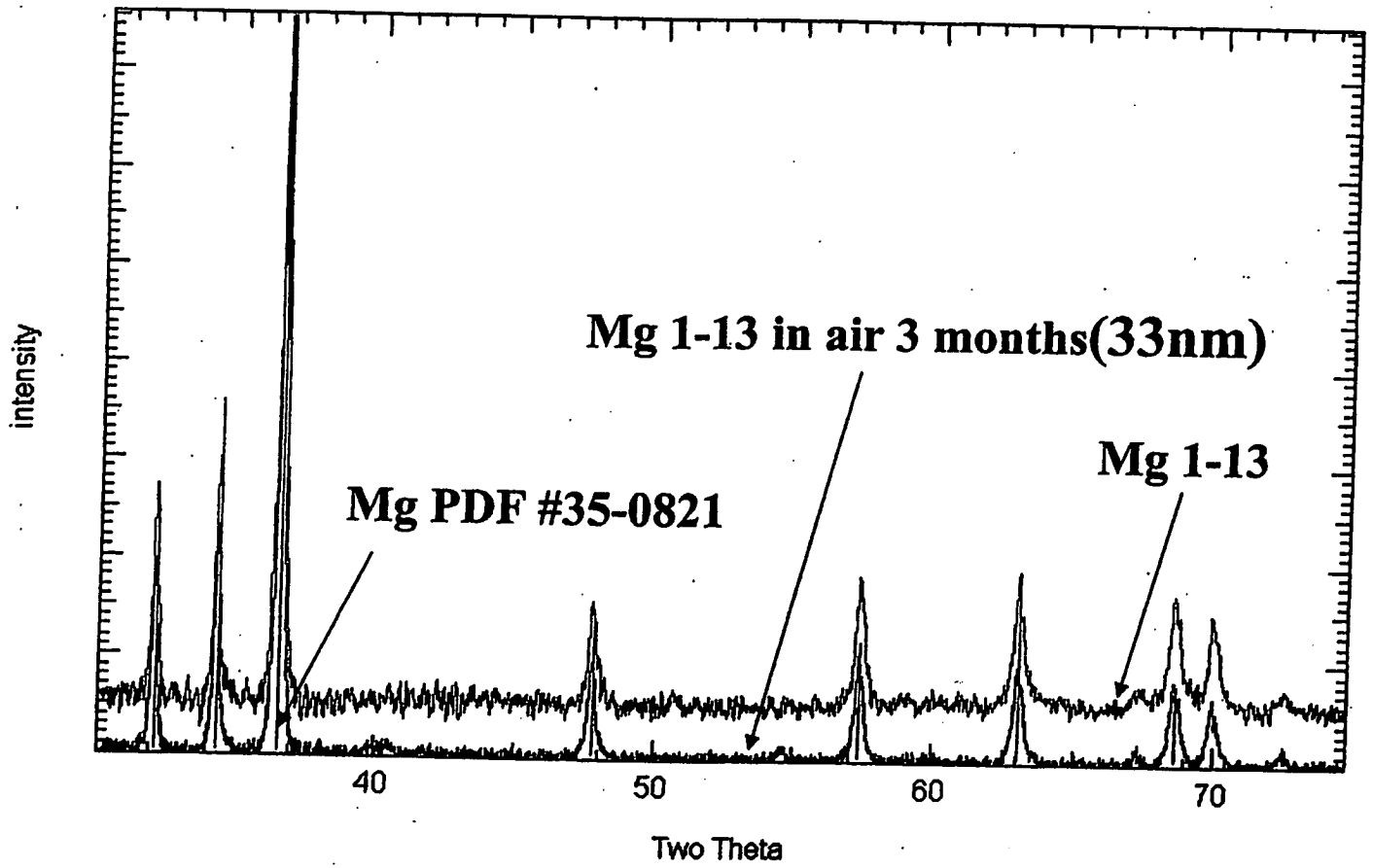


FIG. 5

Phase Stability in Water

After exposure to water for ~48 hours ⇒
Mg 1-13 turned white with XRD giving $Mg(OH)_2$

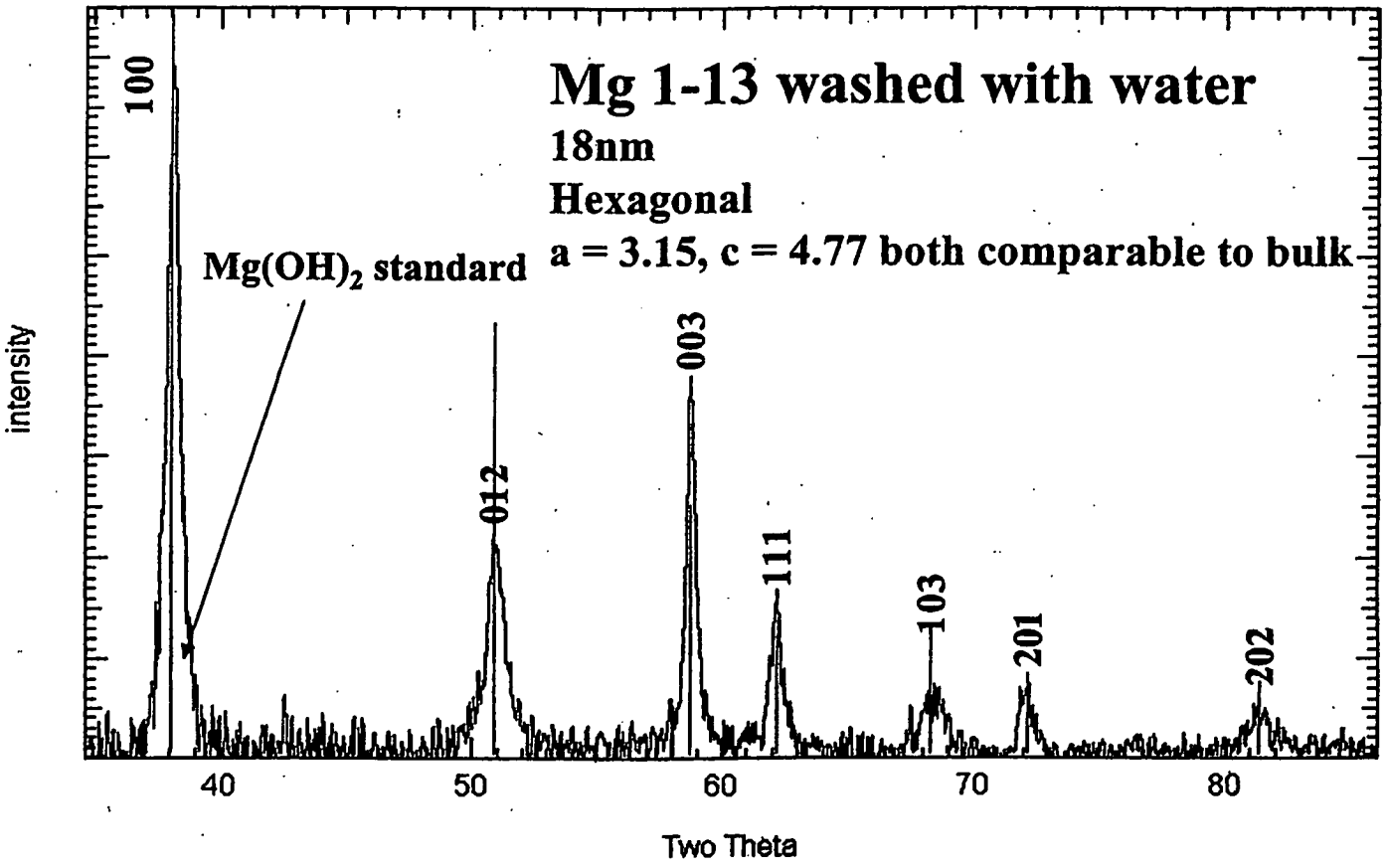


FIG. 6

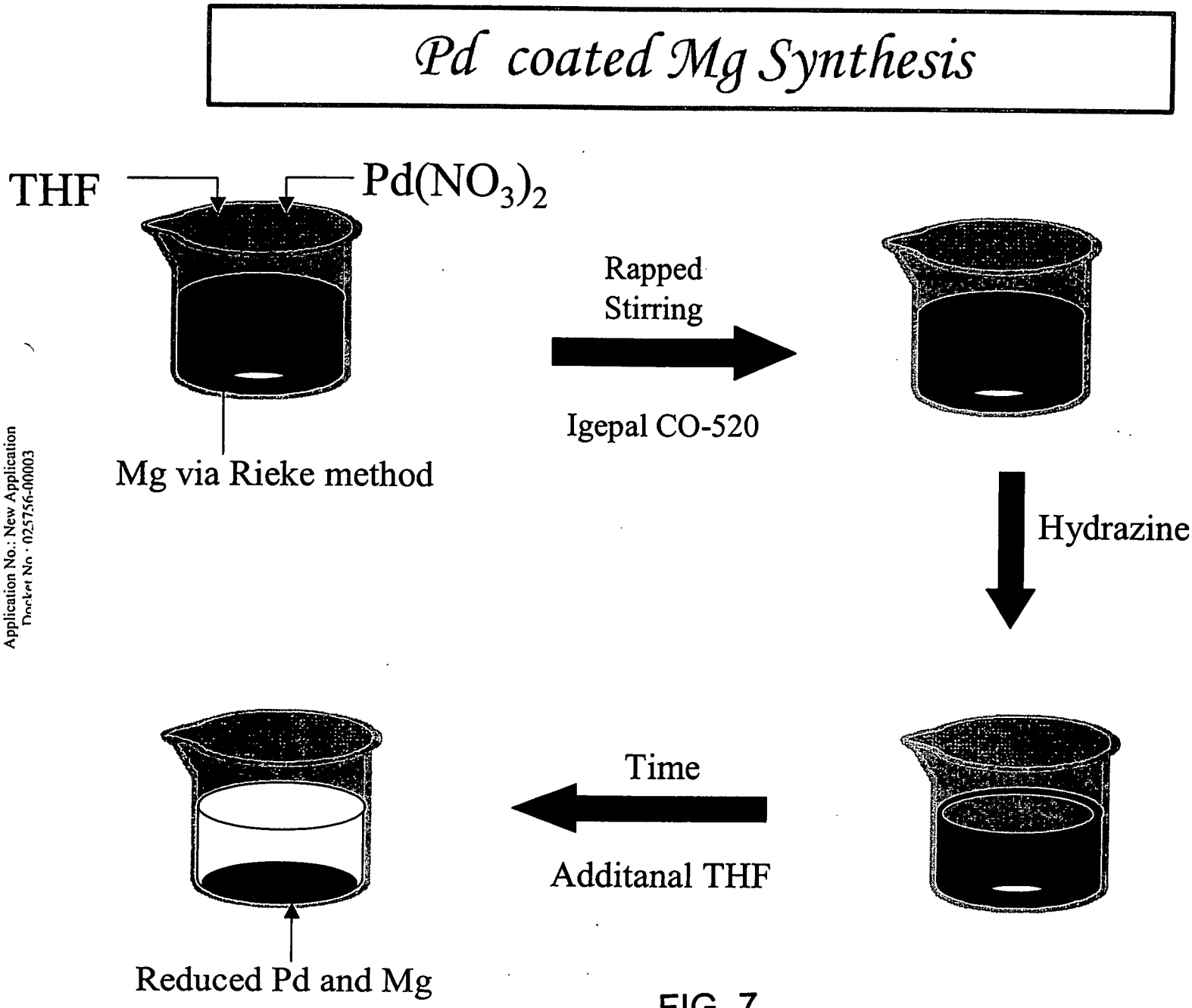


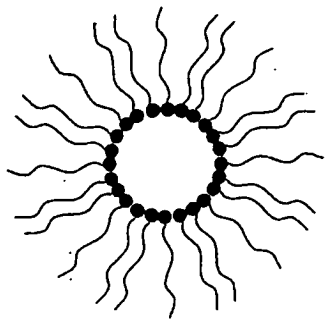
FIG. 7

Spherical nPd

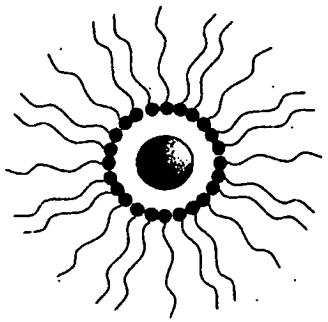
1) Micelle Formation

Igepal™

cyclohexane



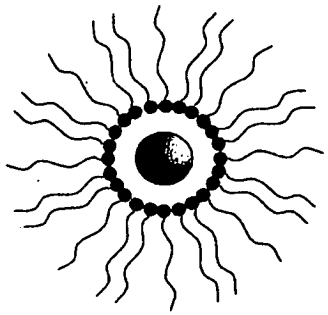
0.04 M Pd in H_2O



2) Reduction of Pd^{+2} using Hydrazine Hydrate ($H_2NNH_2 \cdot xH_2O$)

$$2Pd^{+2}(aq) + N_2H_4(aq) + 4OH^-(aq) \rightarrow 2Pd^0(s) + N_2(g) + 4H_2O$$

3) Break the Micelle and collect the Pd with ethanol



Ethanol

Pd_{nano}

FIG. 8

FIG. 9

Controlling The Size

The size of the Pd spherical particle is dependent on the mole% ratio of surfactant (Igapal) to water often referred to as the R value.

Title: HYDROGEN STORAGE MATERIAL BASED ON A MULTILAYERED CORE/SHELL STRUCTURE
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$$R = [water]/[surfactant]$$

The larger the R value the larger the radius of the particle

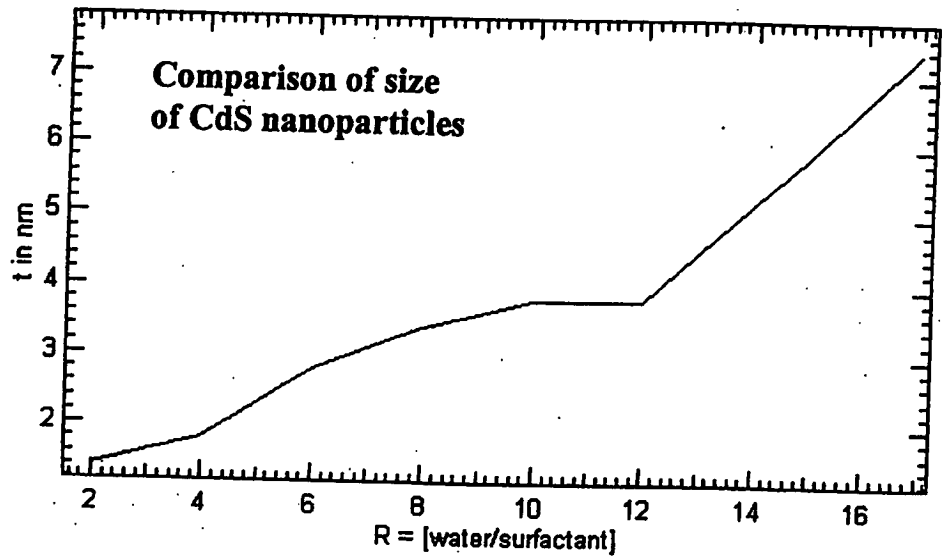


FIG. 10

Spherical Pd Synthesized to Date

Sample	R value	Amount made	Particle size*
Pd 1-62	8	1.3mg	
Pd 1-67	6	4mg	
Pd 1-68-1	1	2mg	
Pd 1-68-2	2	4mg	
Pd 1-69-1	1	.67mg	
Pd 1-68-2	2	1.35mg	
Pd 1-69-6	6	12.3mg	
Pd 1-69-8	8	16.32mg	
Pd 1-70-7	7	19mg	
Pd 1-70-9	9	30.5mg	~9nm
Pd 1-71	9	61mg	
Pd 1-73	5	63.6mg	
Pd 1-74-3	3	61mg	
Pd 1-74-7	7	71mg	
Pd 1-91-6	6	81.4mg	5nm
Pd 1-91-8	8	108.5mg	4nm
Pd 1-96	7	399mg	
Pd 1-100	25	84.8mg	6nm

*By Scherrer equation

DF TEM Spherical Pd

Pd 1-91 R=8; t=4nm

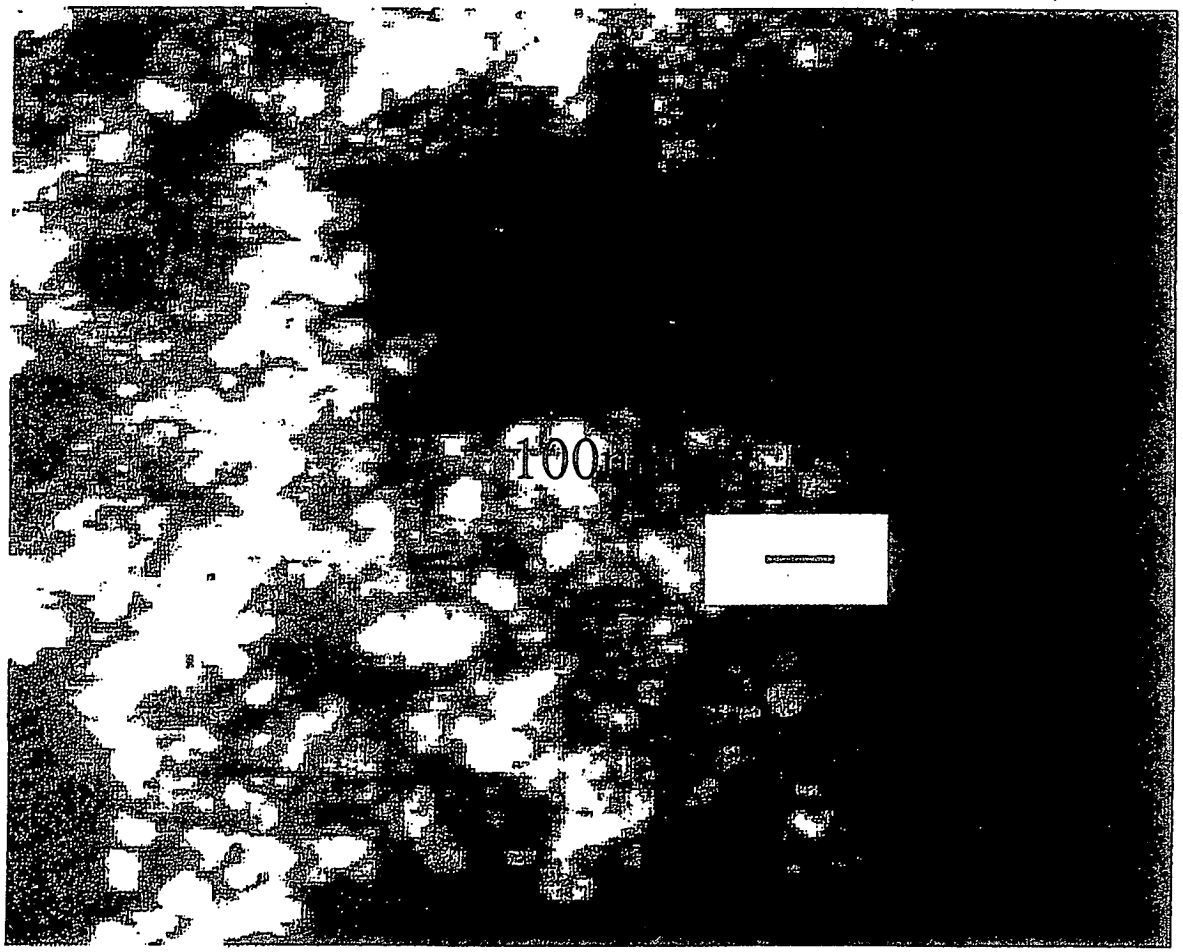


FIG. 11

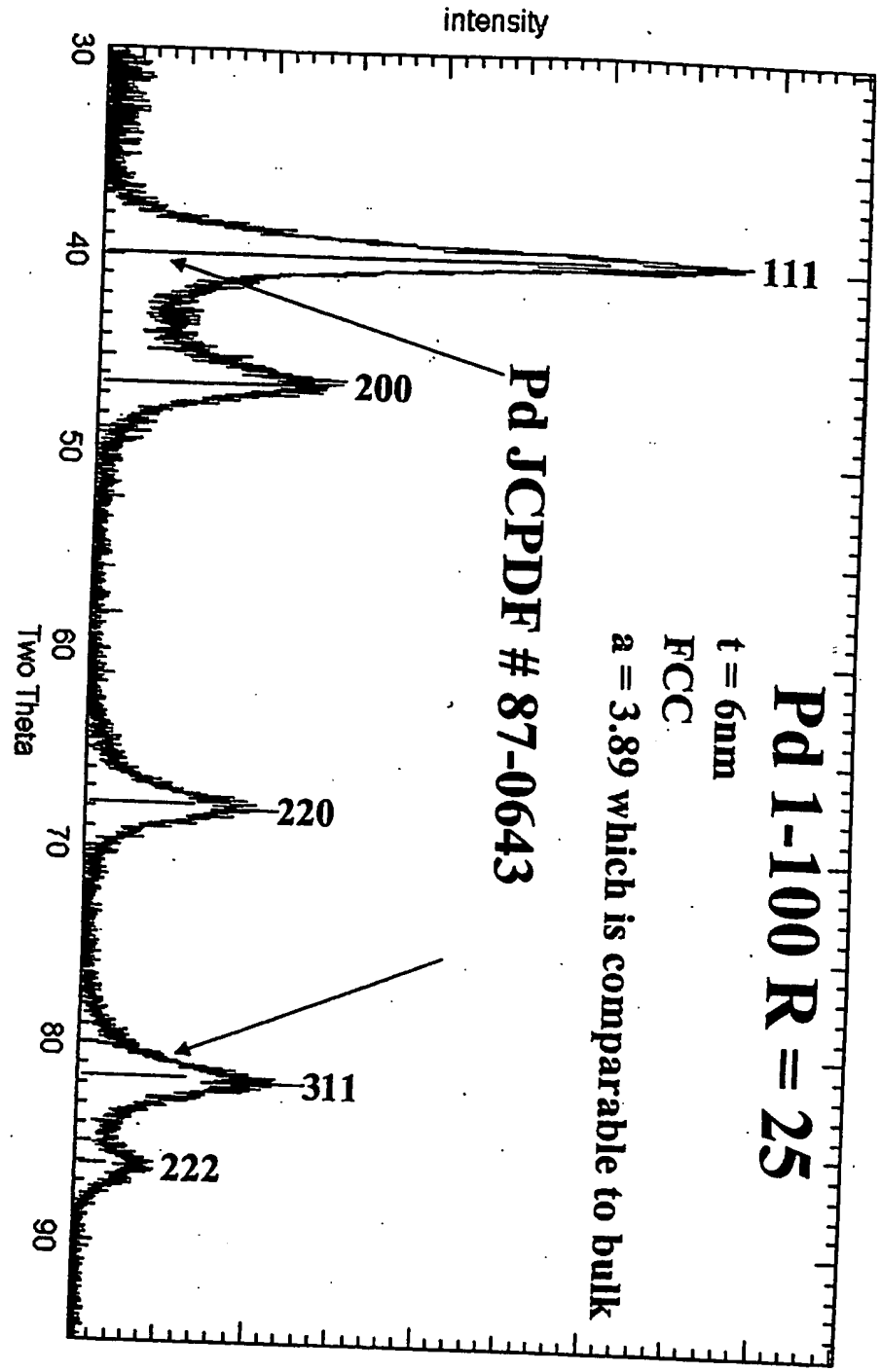
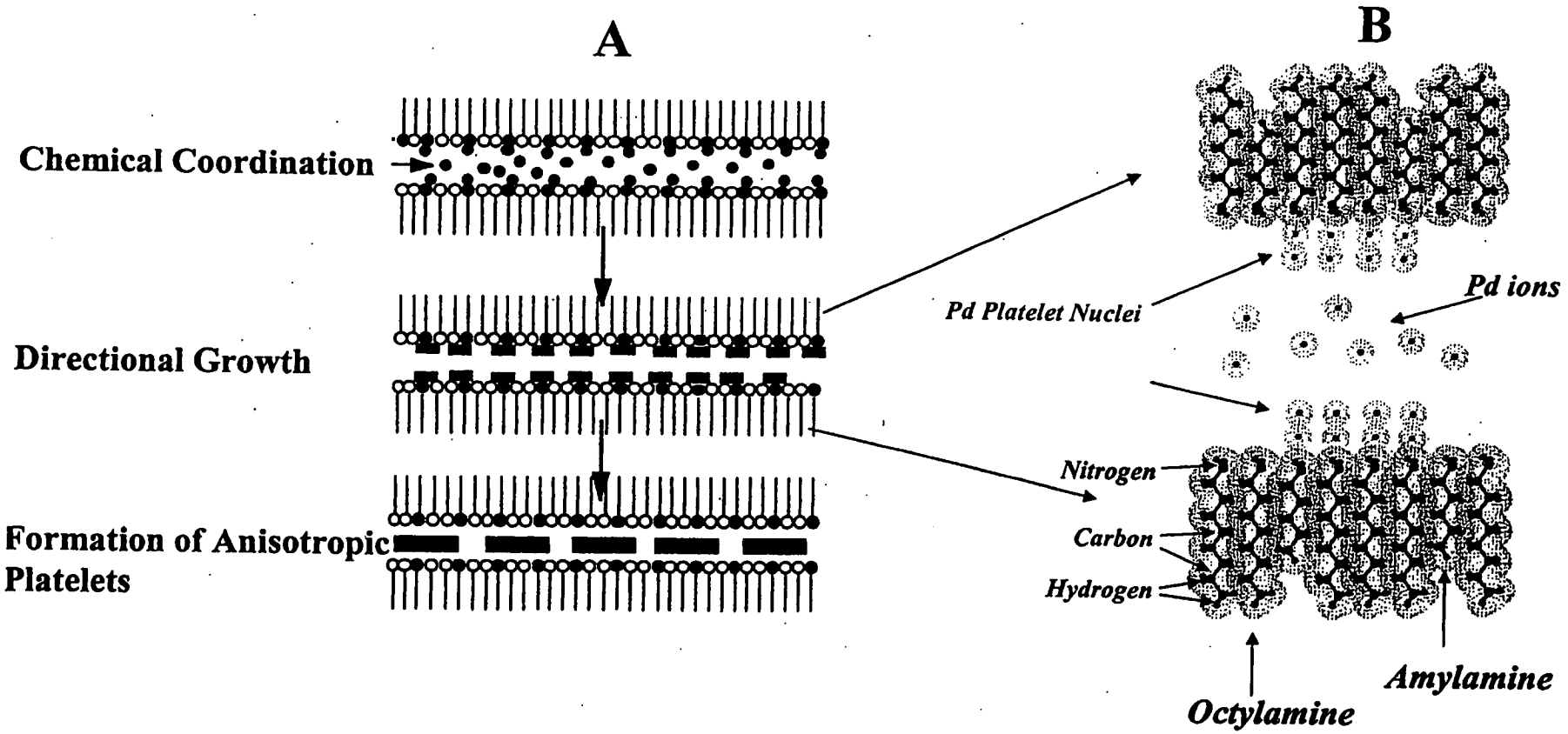

XRD Of Spherical Pd
Pd 1-100 R = 25
t = 6nm
FCC
a = 3.89 which is comparable to bulk
Pd JCPDF # 87-0643
intensity
Two Theta
30
40
50
60
70
80
90
111
200
220
311
222

FIG. 12

FIG. 13

Synthetic Approach For Platelet Pd Particles



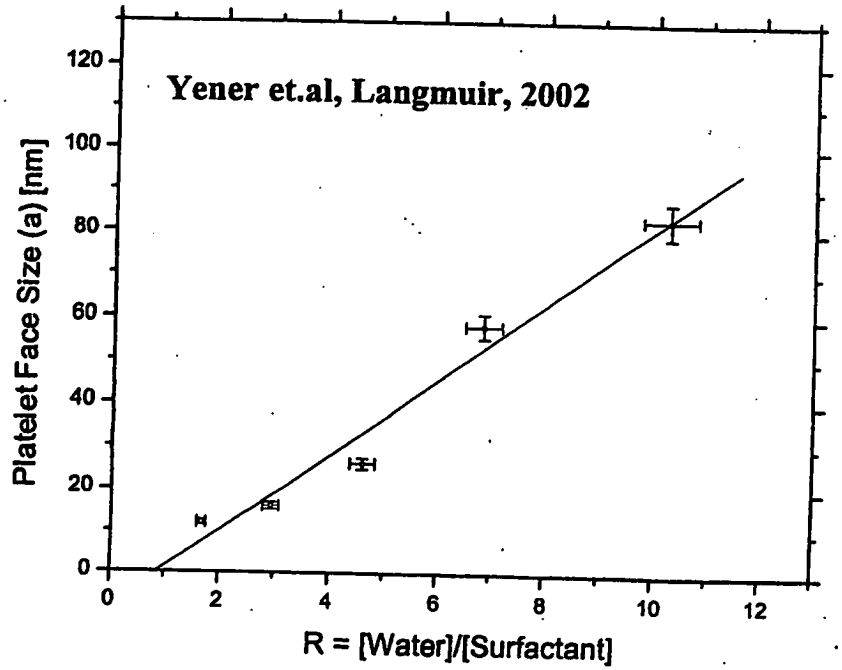
Platelet Size Control

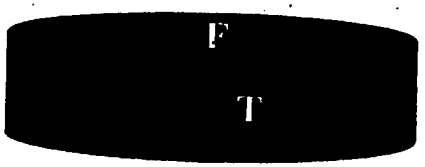
R = [Water]/[Surfactant]

Surfactant = Octylamine + 5% Amylamine

$[CH_3(CH_2)_7NH_2]$ $[CH_3(CH_2)_4NH_2]$

The platelet face size (a) as a function of R.





F = Face

T = Thickness

FIG. 14

Platelets Made

Sample	R Value	Amount Made	F*	T*
Pd 1-75	6.8	20.4mg		
Pd 1-77	20.4	228mg		
Pd 1-80-3.4	3.4	66mg		
Pd 1-80-1.7	1.7	33mg		
Pd 1-86	10.2	236mg		
Pd 1-91-8.5	8.5	212mg		
Pd 1-91-6.8	6.8	254mg		
Pd 2-5-1.7	1.7	21.2		
Pd 2-5-3.4	3.4	21.2		
Pd 2-5-6.8	6.8	21.2	33nm	2nm
Pd 2-5-8.5	8.5	21.2		
Pd 2-5-10.2	1.2	21.2	150nm	6nm
Pd 2-5-20.4	20.4	21.2		
Pd 2-8a	10.2	21.2	130nm	8nm
Pd 2-8b (AA)	10.2	21.2	80nm	1.7nm

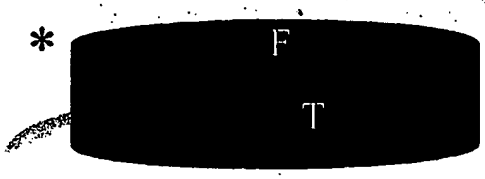


FIG. 15

nMg Materials Synthesized

Sample	Amount Made
Mg 1-2	0.198g
Mg 1-10	0.4g
Mg 1-13	0.4g
Mg 1-18	0.2g
Mg 1-41	0.2g
Mg 1-51	0.4g
Mg 1-57a	0.5g
Mg 1-57b	0.5g
Mg 1-57c	0.5g

Platelet nPd

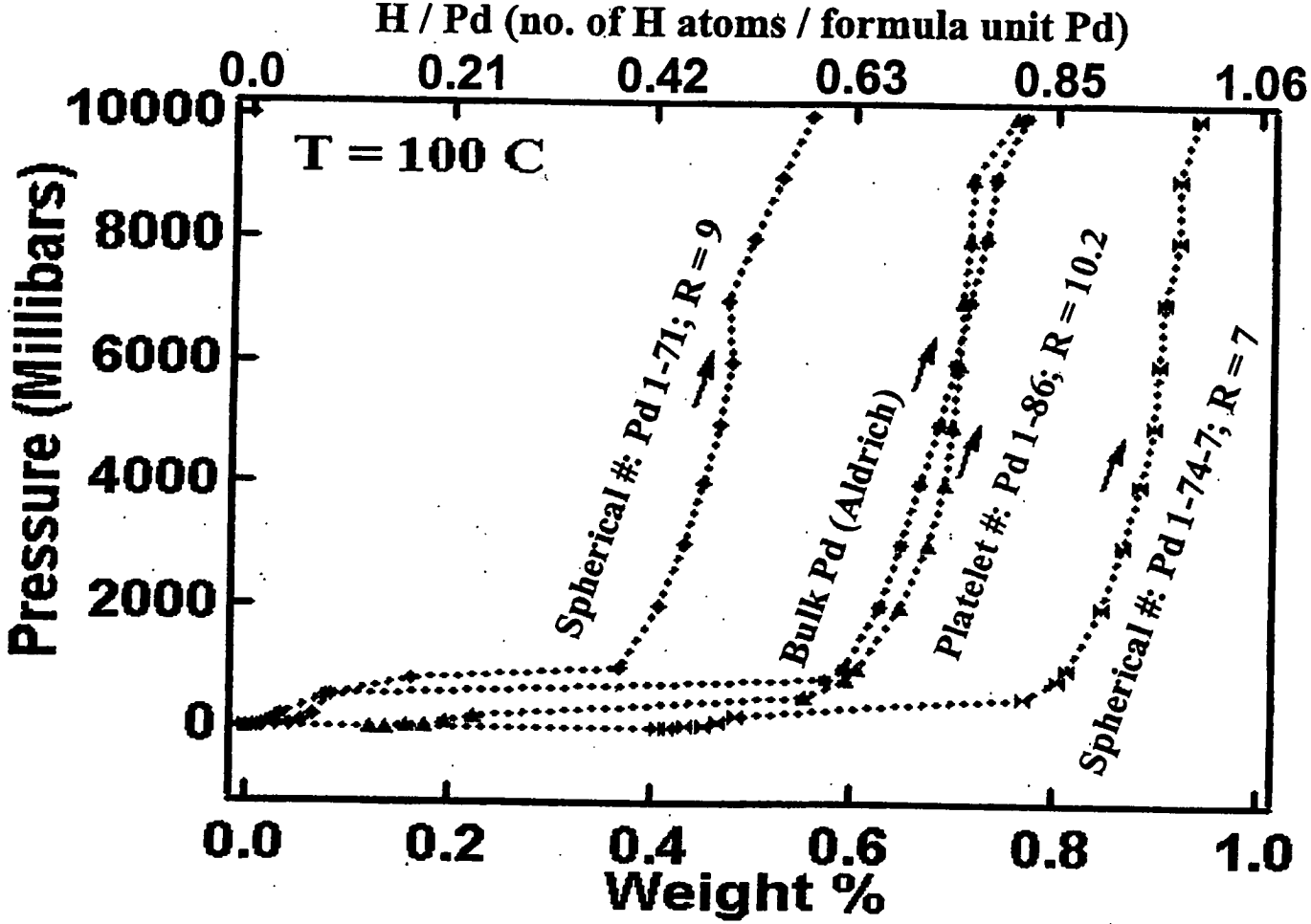
Sample	R Value	Amount Made
Pd 1-75	6.8	20.4mg
Pd 1-77	20.4	228mg
Pd 1-80-3.4	3.4	66mg
Pd 1-80-1.7	1.7	33mg
Pd 1-86	10.2	236mg
Pd 1-91-8.5	8.5	212mg
Pd 1-91-6.8	6.8	254mg
Pd 2-5-1.7	1.7	21.2
Pd 2-5-3.4	3.4	21.2
Pd 2-5-6.8	6.8	21.2
Pd 2-5-8.5	8.5	21.2
Pd 2-5-10.2	1.2	21.2
Pd 2-5-20.4	20.4	21.2
Pd 2-8a	10.2	21.2
Pd 2-8b (AA)	10.2	21.2

Spherical nPd

Sample	R Value	Amount Made
Pd 1-62	8	1.3mg
Pd 1-67	6	4mg
Pd 1-68-1	1	2mg
Pd 1-68-2	2	4mg
Pd 1-69-1	1	.67mg
Pd 1-68-2	2	1.35mg
Pd 1-69-6	6	12.3mg
Pd 1-69-8	8	16.32mg
Pd 1-70-7	7	19mg
Pd 1-70-9	9	30.5mg
Pd 1-71	9	61mg
Pd 1-73	5	63.6mg
Pd 1-74-3	3	61mg
Pd 1-74-7	7	71mg
Pd 1-91-6	6	81.4mg
Pd 1-91-8	8	108.5mg
Pd 1-96	7	399mg
Pd 1-100	25	84.8mg

FIG. 16

Adsorption Isotherms of Palladium NanoParticles and Bulk Sample at 100 °C



Bulk sample particle size: 1.0 – 1.5 Microns
Spherical particles: **R = 7** (5nm) and **R = 9(8nm)**;
Platelets **R=10.2 (8nm thick)**

FIG. 17

Title: HYDROGEN STORAGE MATERIAL
BASED ON A MULTILAYERED CORE/SHELL
STRUCTURE
Inventor's Name: Peter C. EKLUND, et al.
Application No.: New Application
Docket No.: 025756-00003

Comparison of TGA Evaluations at 100C for Nano and Bulk Samples

Sample	Knee for H/Pd Ratio	Plateau Onset (Wt %)	Estimated Adsorption Rate* (H/Pd/min)	Estimated Desorption Rate* (H/Pd/min)
Pd 1-74-7 R = 7 (4-5 nm Spheres)	~ 0.78	~ 0.47	~0.01	~0.005
Pd 1-86 R = 10.2 (8 nm thick Platelets)	~ 0.65	~ 0.2	0.007	0.003
Bulk (1.0 – 1.5μm)	~ 0.6	~ 0.08	0.005	0.002

* Possibly affected by sintering

FIG. 18

Title: HYDROGEN STORAGE MATERIAL BASED ON A MULTILAYERED CORE/SHELL STRUCTURE
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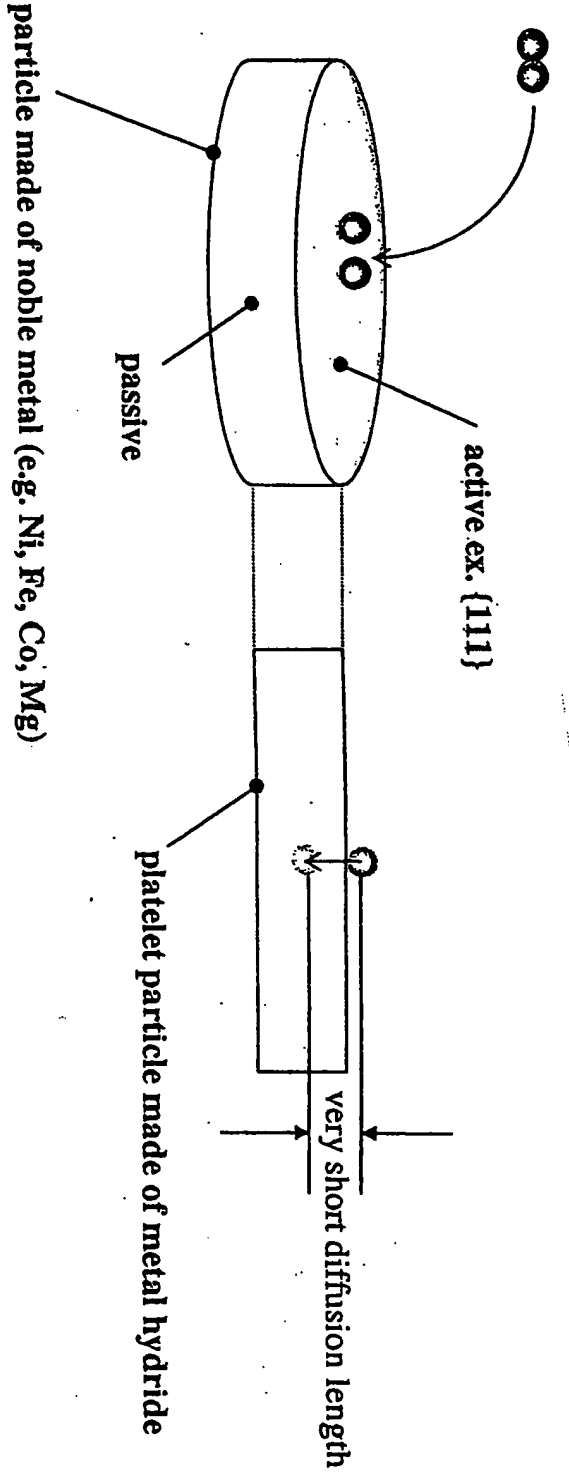


FIG. 19

FIG. 20

FIG. 21

TGA Compositional Analysis

Sample	Pd in sample	Wt.%Pd as PdO in sample	Wt.% Carbon	η corr. factor
Spherical				
Pd 2-35 R=2 (4nm)	95.34%	0%	2.37%	1.05
Pd 2-71 R=8	96.16%	0%	0.05%	1.04
Platelet				
Pd 2-65-10 R=10 (327nm X 2.8nm to 109nm X 1.5nm)	87.26%	0%	0.08%	1.15
Pd 2-65-7 R=7 (82nm X 1.3nm)	94.69%	0%	0.17%	1.06

FIG. 22 **NanoCrystalline vs NanoParticle**

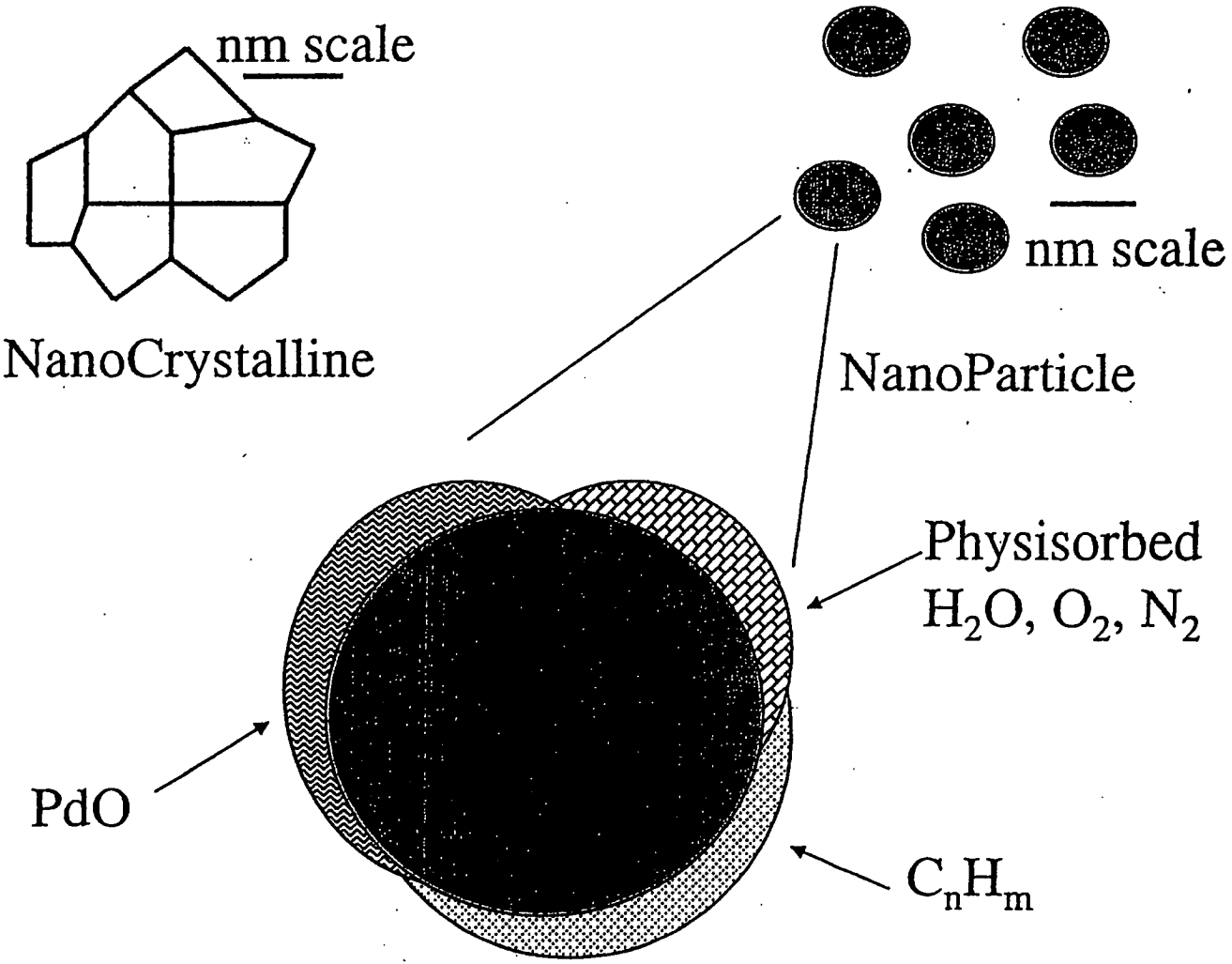
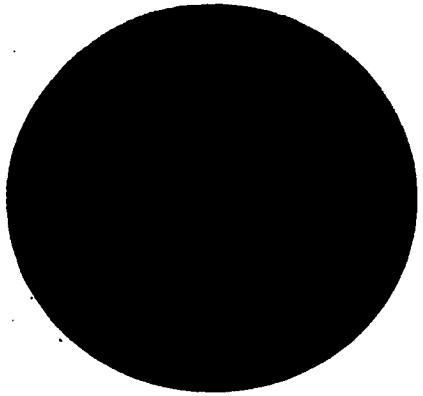


FIG. 23 . **Effect of morphology on particle packing**

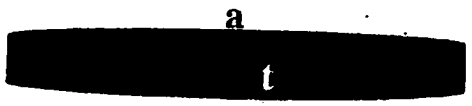
Spherical



r ~ 3-50 nm

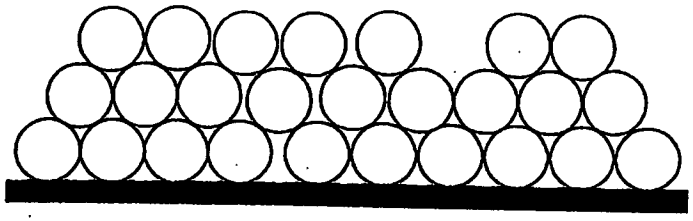
VS

Platelet



t ~ 1-200 nm
a/t ~ 10-100

Particle Packing Characteristics



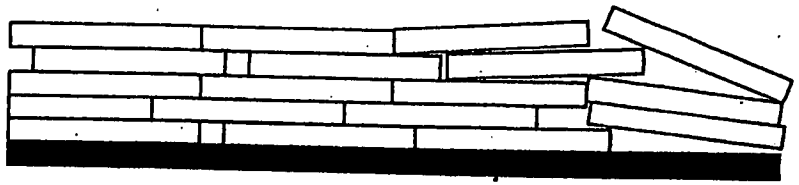


FIG. 24 **HRTEM of Platelet nanoPd**

Moire fringes

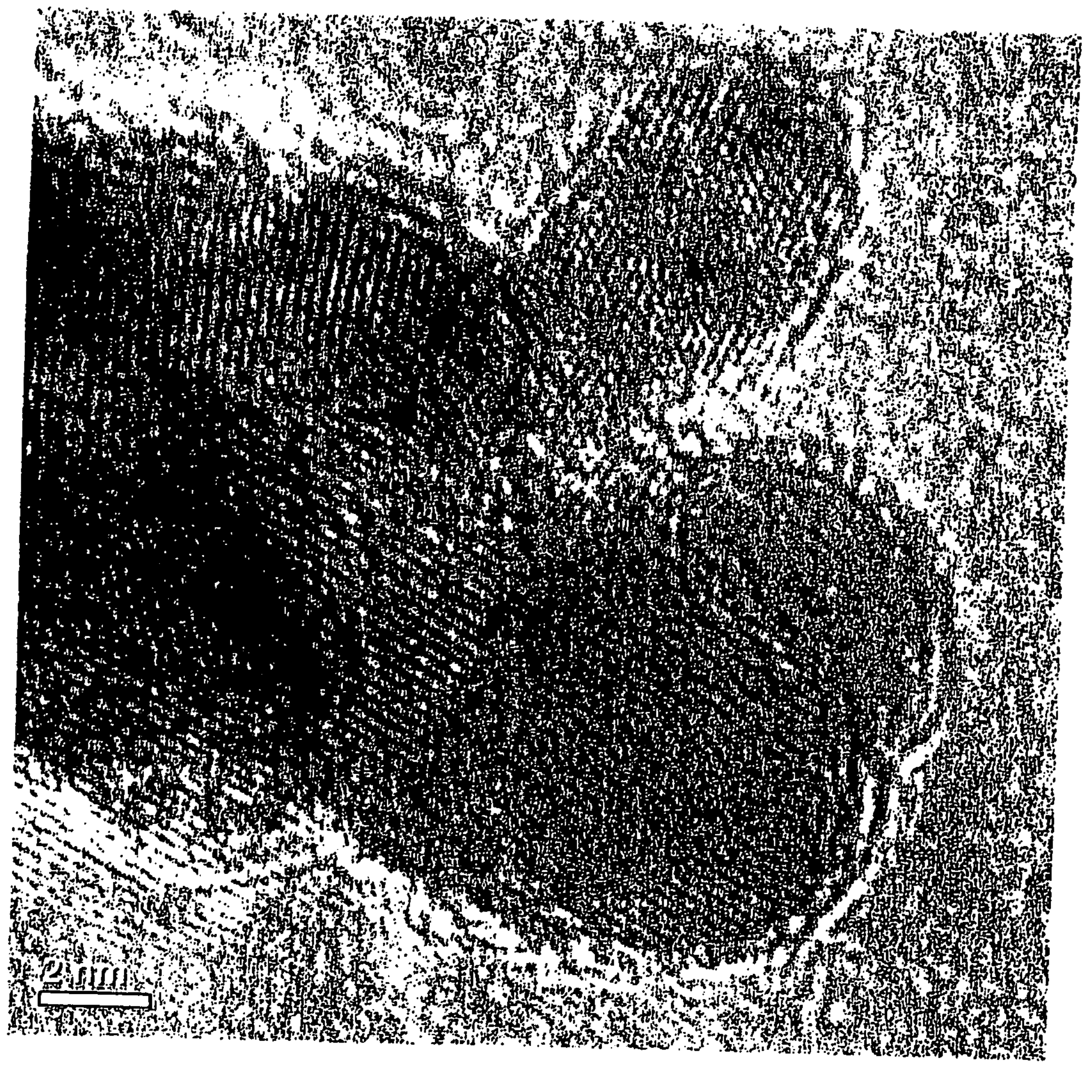
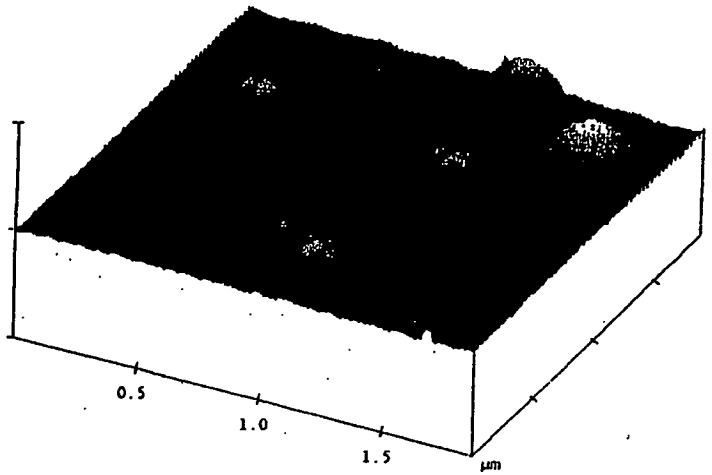
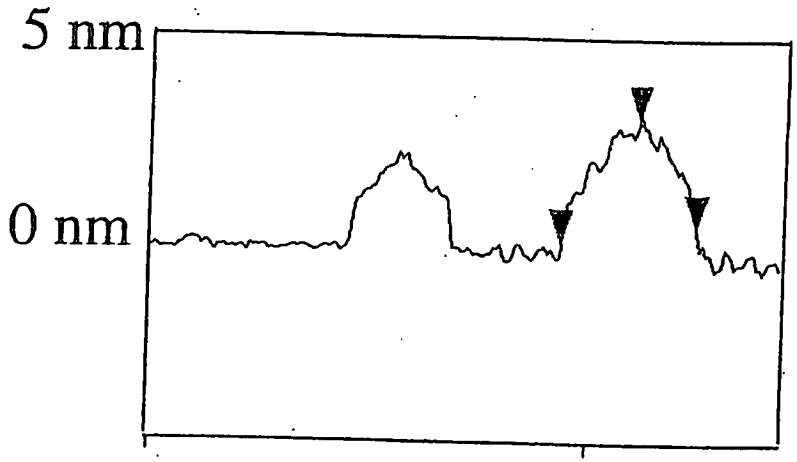
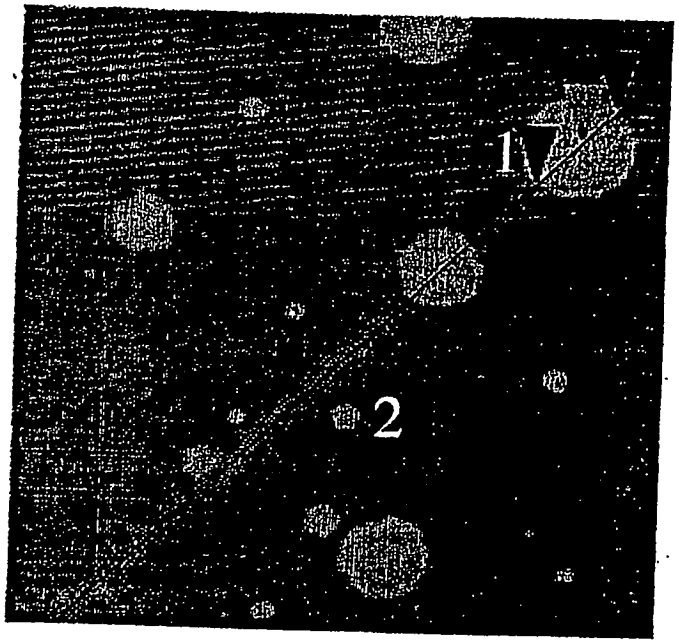


FIG. 25

AFM of Platelet nanoPd







(1) 327nm X 2.8nm
(2) 109nm X 1.5nm

Title: HYDROGEN STORAGE MATERIAL BASED ON A MULTILAYERED CORE/SHELL STRUCTURE
Inventor's Name: Peter C. EKLUND, et al.
Application No.: New Application
Docket No.: 025756-00003

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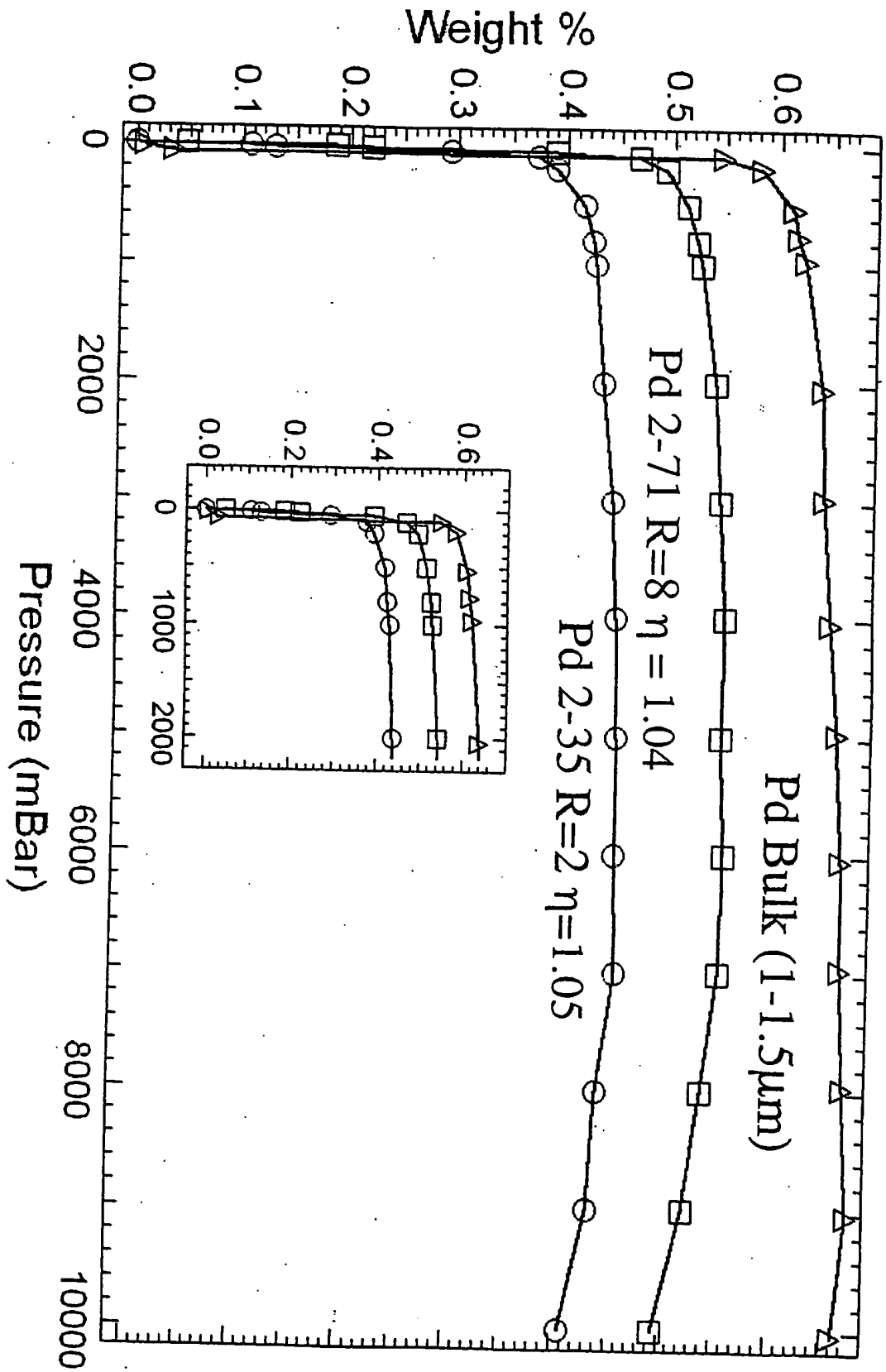
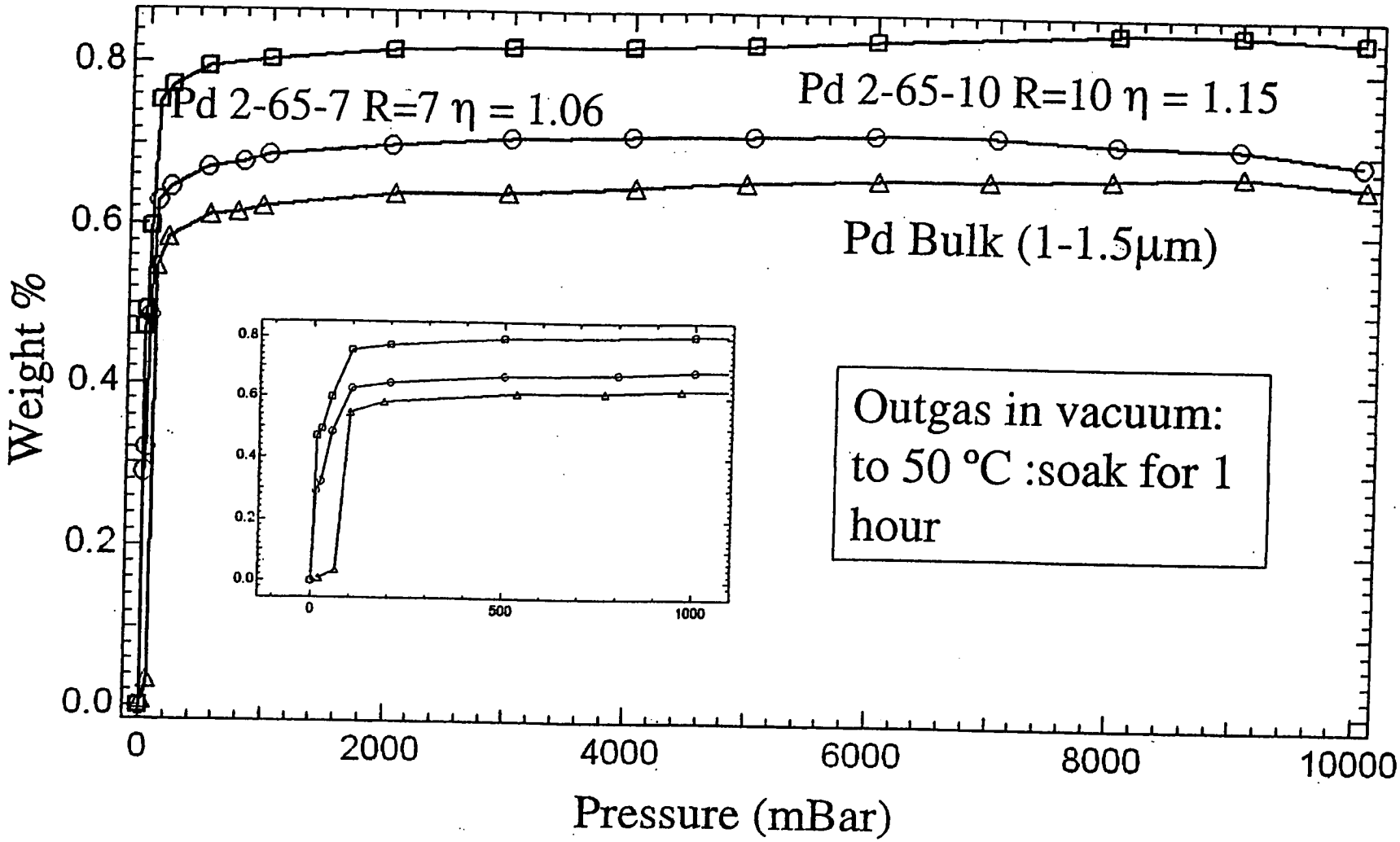


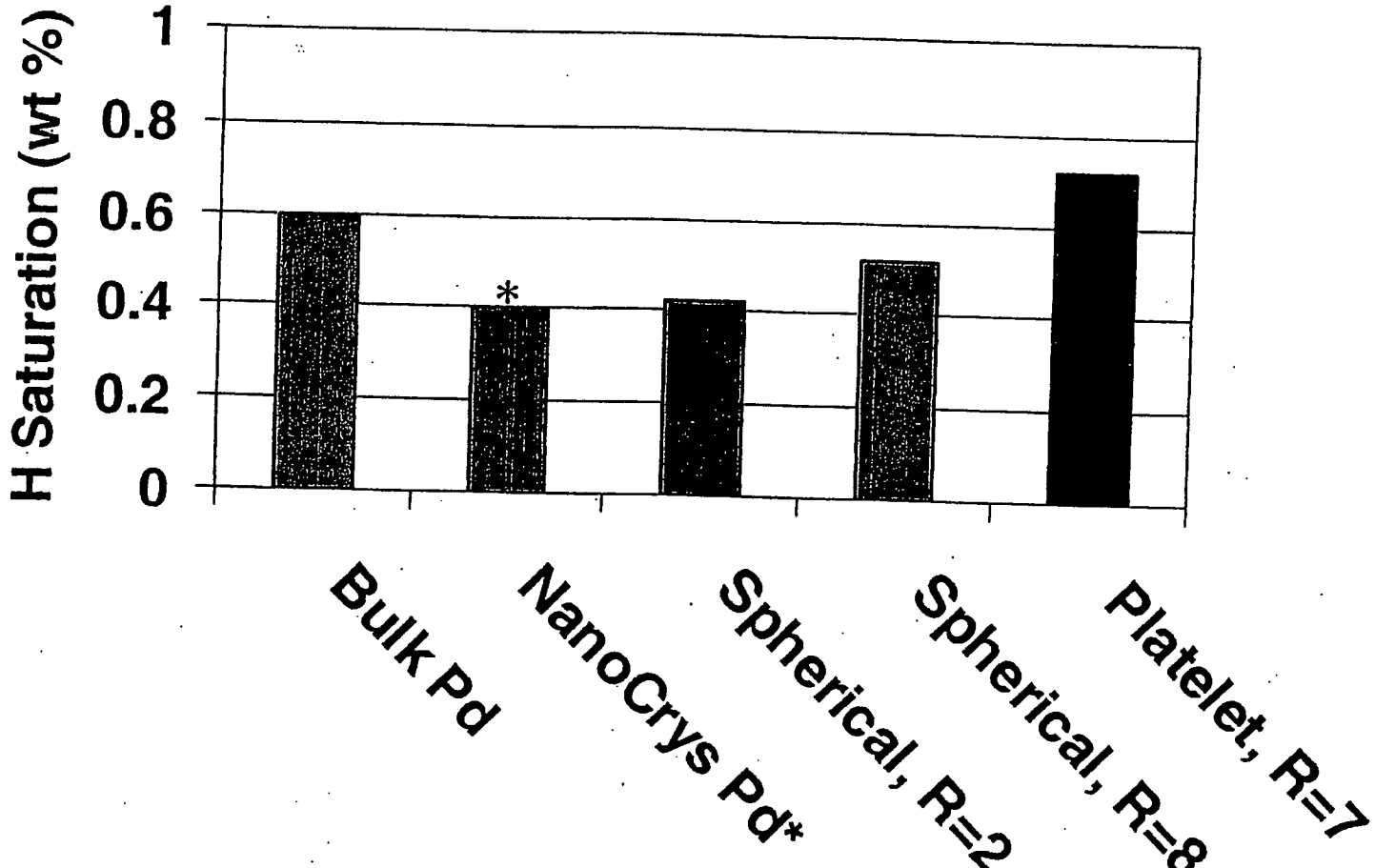
FIG. 26 H Adsorption of Spherical Pd

FIG. 27 H Adsorption of Pd Platelets



Title: HYDROGEN STORAGE MATERIAL BASED ON A MULTILAYERED CORE/SHELL STRUCTURE
Inventor's Name: Peter C. EKLUND, et al.
Application No.: New Application
Docket No.: 025756-00003

FIG. 28 **Hydrogen Saturation vs Pd Material**



* Kuji, et.al, taken as maximum H/Pd at 0.1 MPa at 353K

Title: HYDROGEN STORAGE MATERIAL
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STRUCTURE
Inventor's Name: Peter C. EKLUND, et al.
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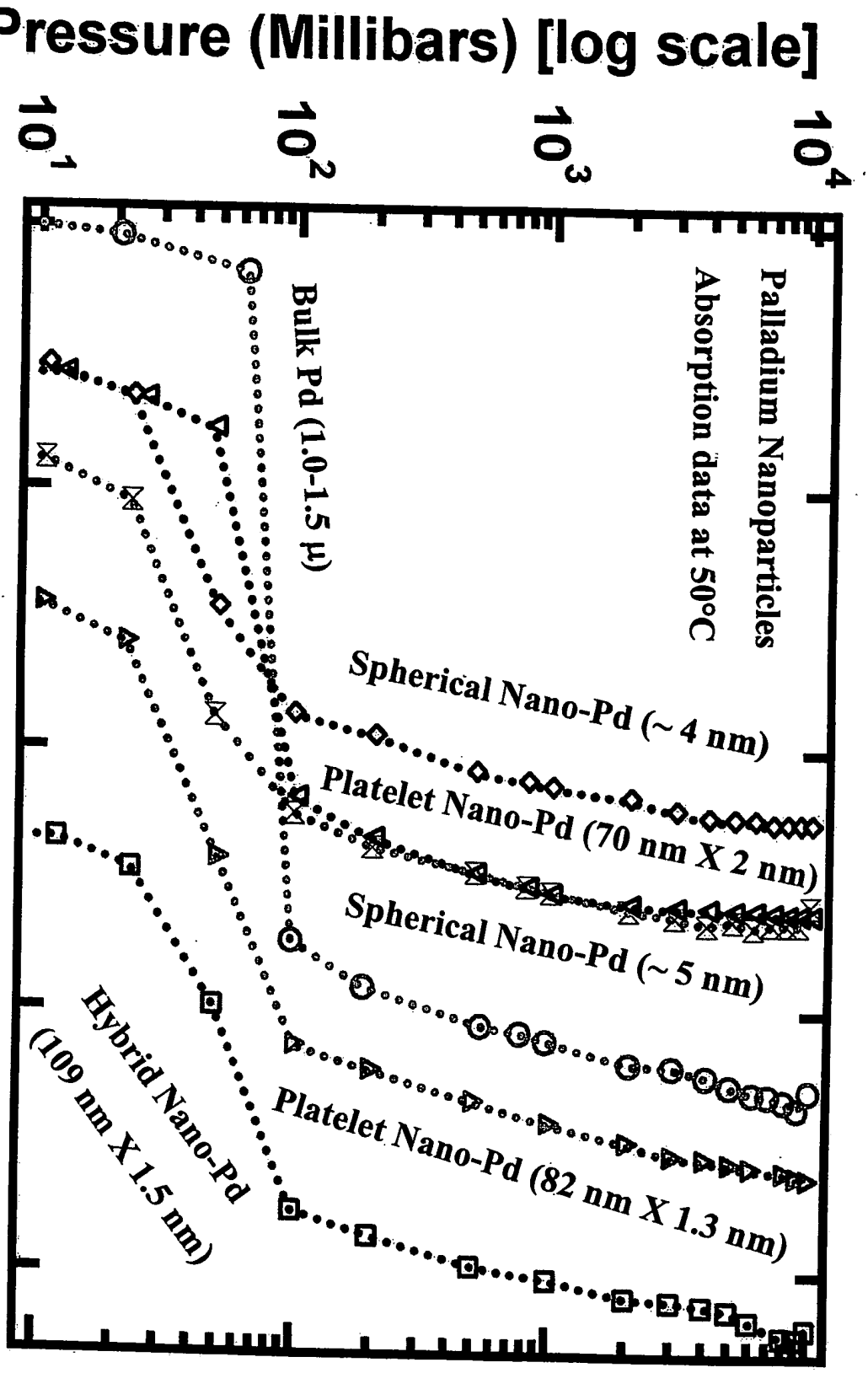


FIG. 29

FIG. 30
HRTEM of Platelet Particles

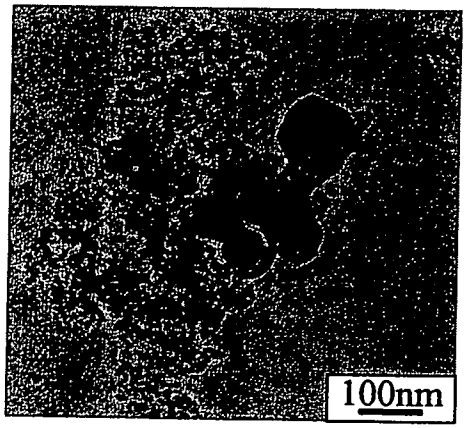
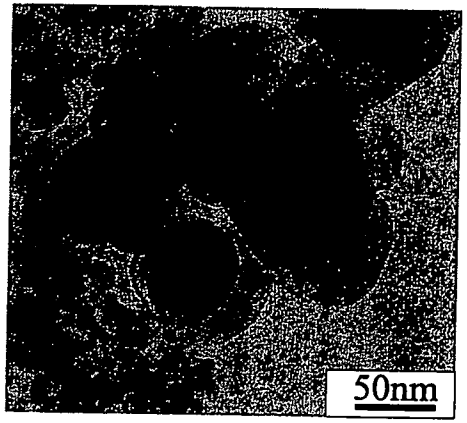


Fig 30a



Mixed (hybride) Platelet Sample Fig 30b

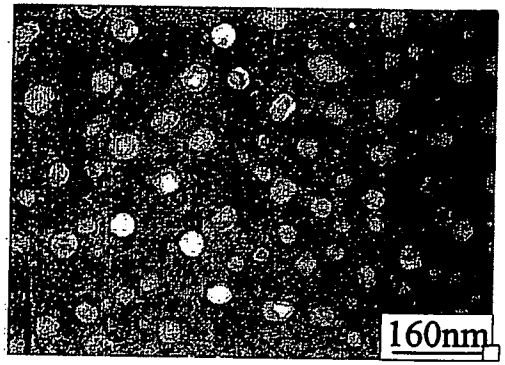


Fig 30c

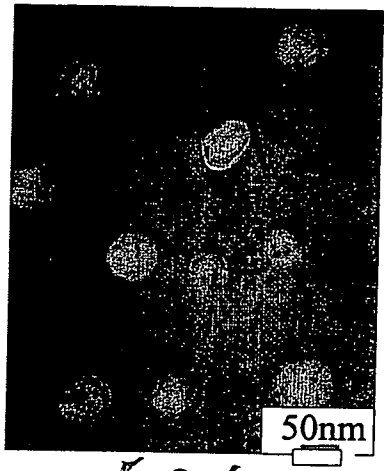


Fig 30d

Platelet Sample average size 70nm X 1.7nm

Title: HYDROGEN STORAGE MATERIAL BASED ON A MULTILAYERED CORE/SHELL STRUCTURE
Inventor's Name: Peter C. EKLUND, et al.
Application No.: New Application
Docket No.: 025756-00003

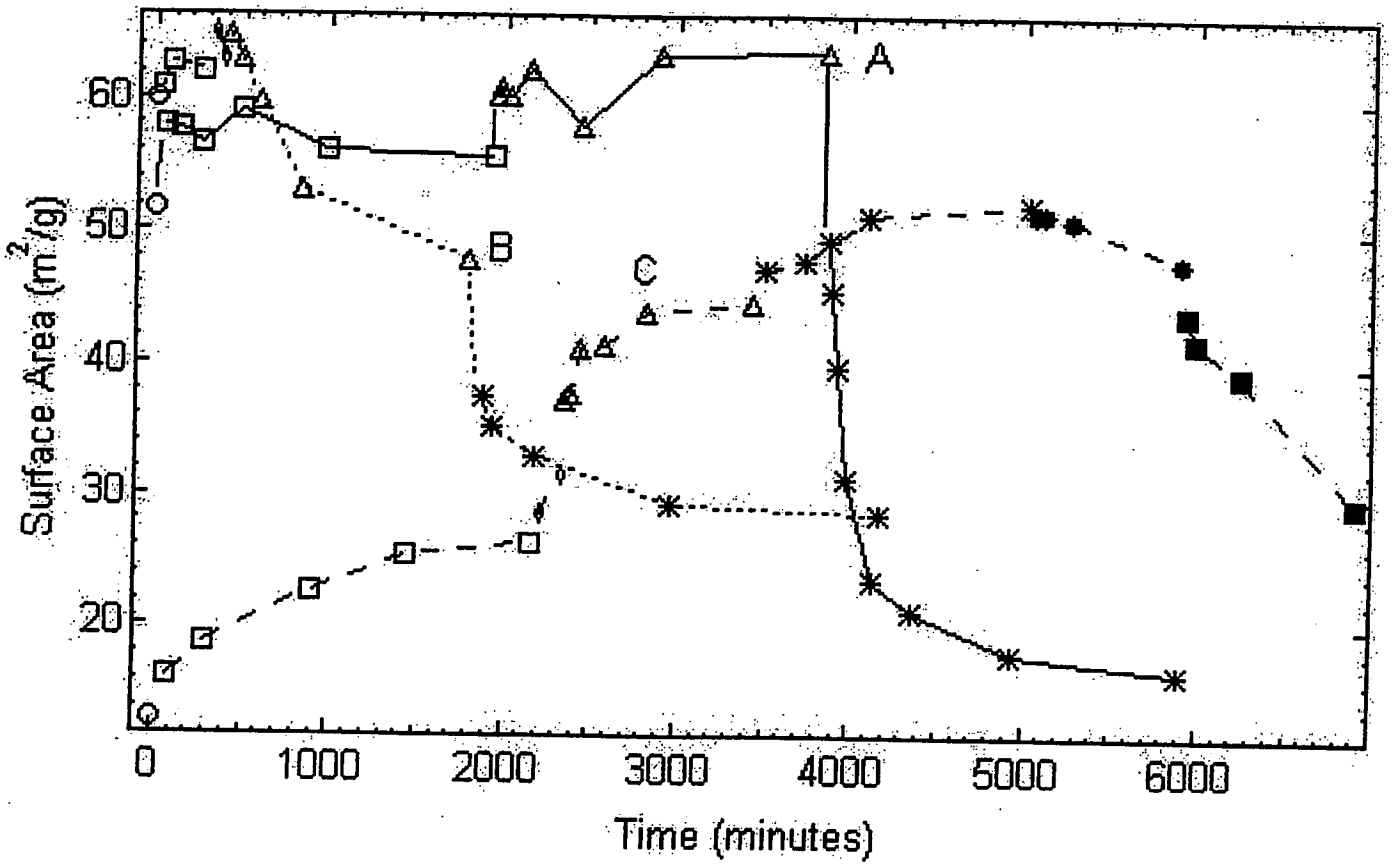
Review of nanoPd Sample tested for Hydrogen Storage

Sample		R	%Pd	d[1]	a[2]	t[3]
Reverse Micelle Synthesis						
2-35*	spherical	2	95	4		
2-71*	spherical	8	96	5		
2-44	spherical	5	82			
Bilayer Synthesis						
2-65-10	mixed	10	87	5	327	2.8
					109	1.5
3-18	platelet	8	96		70	2
3-33	platelet	6	94		130	2.6

R = [water]/[Surfactant], 1= diameter of spherical particle, 2 = face size of platelet particle, and 3 = thickness of platelet particle in nm. * Samples washed with hydrazine

FIG. 31

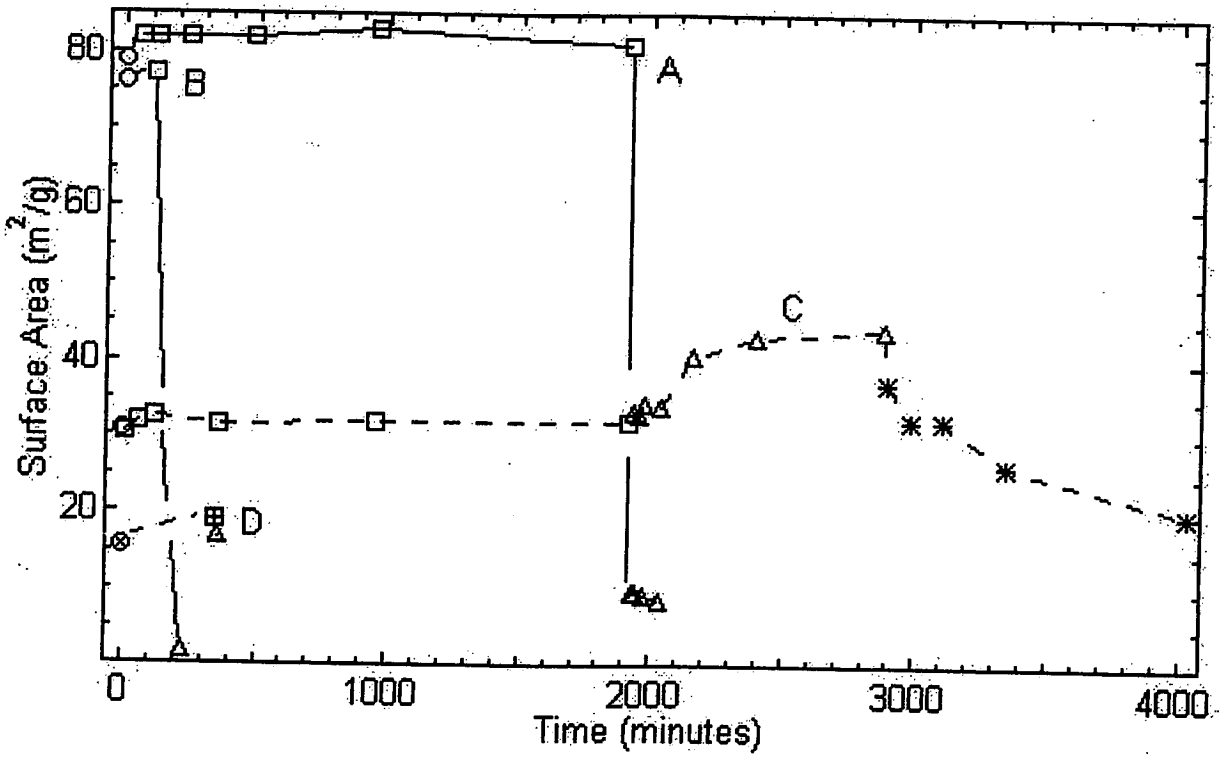
Sintering of Platelet Particles



Surface area vs. heating time for sample synthesized by bilayers. Samples A(sample 3-18) and B(sample 3-33) consist of only platelets. Sample C(sample 2-65-10) is a mix of platelets and spherical particles. Heating temperatures are O = room temperature, □ = 50 °C, ◇ = 75 °C, △ = 100 °C, ✳ = 150 °C, ● = 200 °C. ■ = 250 °C.

FIG. 32

Sintering of Spherical Particles



Surface area vs. heating time for spherical particles synthesized via reverse micelles. A(sample 2-44) and B(sample 2-35) = not washed with hydrazine hydrate. C(sample 2-71) and D(sample 2-35) = washed with hydrazine hydrate. Lines B and D are both of sample 2-35. Heating temperatures are O = room temperature, □ = 50 °C, △ = 100 °C, and ✱ = 150 °C.

FIG. 33

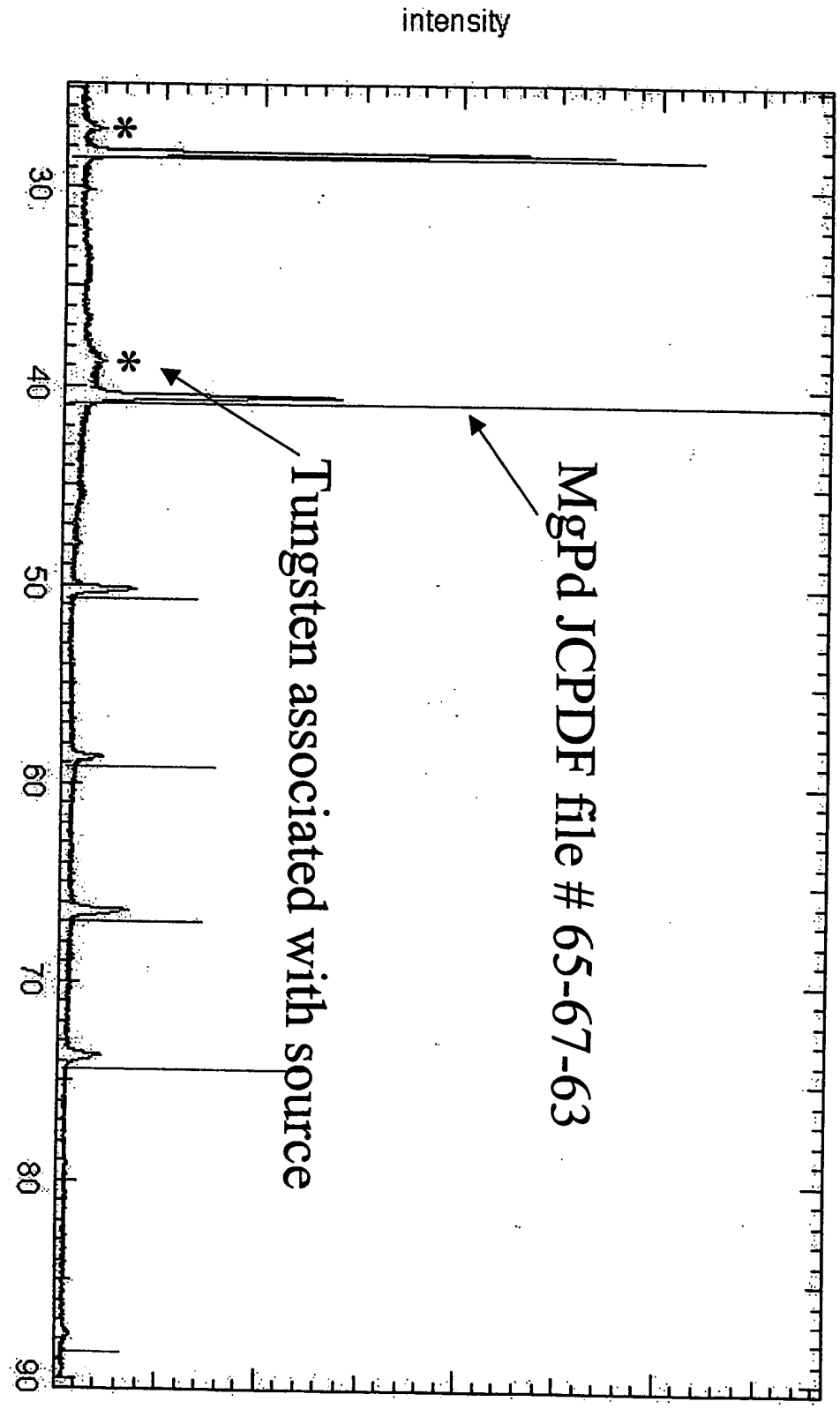
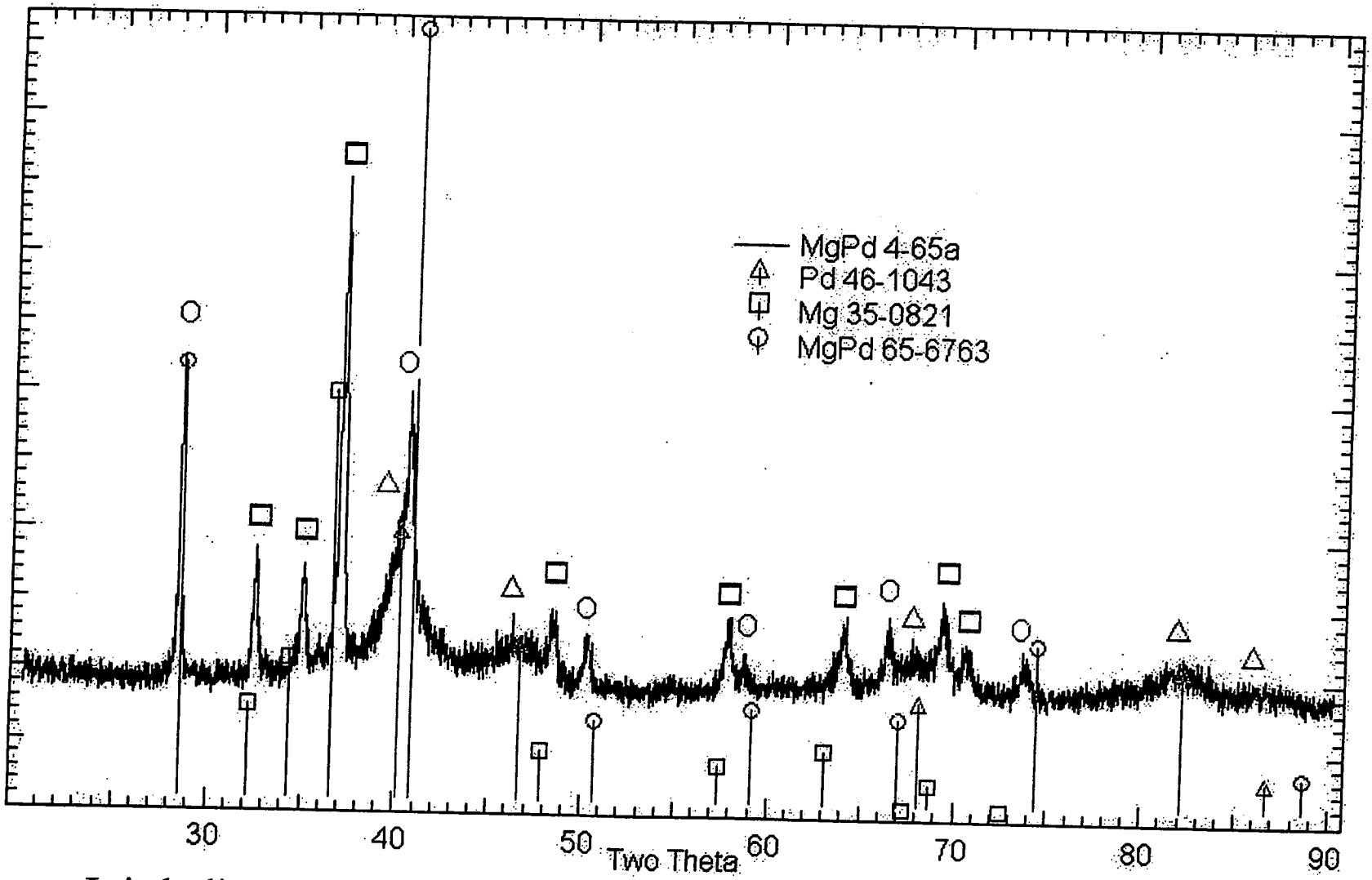

intensity
30
40
50
60
70
80
90
Two Theta
*
*
Tungsten associated with source
MgPd JCPDF file # 65-67-63
MgPd Alloy

FIG. 34

MgPd Alloy with Mg and Pd Metal



It is believed that excess Mg in the reaction lead to the formation of MgPd alloy along with metallic Pd and Mg.

FIG. 35